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IS 7647 (2003): Acetoacet-2-Toluidide [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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“Knowledge is such a treasure which cannot be stolen”



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भारतीय मानक  
ऐसिटोऐसिट-2-टोल्युडाईड—विशिष्टि  
( पहला पुनरीक्षण )

*Indian Standard*  
ACETOACET-2-TOLUIDIDE—SPECIFICATION  
( *First Revision* )

ICS 71.080.80

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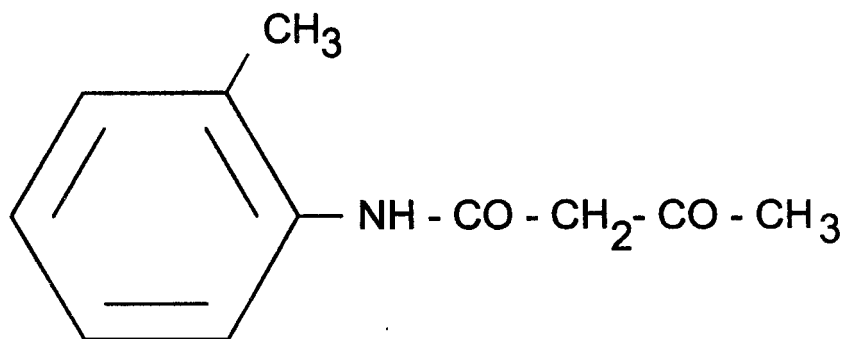
**BUREAU OF INDIAN STANDARDS**  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
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## FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dyes Intermediate Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1975 and has been revised in the light of experience gained during the long span of period. In the present revision, thin layer chromatography has been included as the method of test to keep pace with the present trends in the industry.

Acetoacet-2-toluidide ( $C_{11}H_{13}NO_2$ ) is an important dye intermediate, used in the manufacture of yellow organic pigments. It is represented by the following structural formula:



Acetoacet-2-Toluidide  
(Molecular Mass: 191.2)  
CAS Registry No. (93-68-5)

The composition of the Committee responsible for formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

# Indian Standard

## ACETOACET-2-TOLUIDIDE—SPECIFICATION

### ( First Revision )

#### 1 SCOPE

This standard prescribes the requirements and methods of sampling and test for Acetoacet-2-toluidide.

#### 2 REFERENCES

The following standards contain provisions which through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards given below:

<i>IS No.</i>	<i>Title</i>
1070 : 1992	Reagent grade water ( <i>third revision</i> )
2552 : 1989	Steel drums (galvanized and ungalvanized) ( <i>third revision</i> )
5299 : 2001	Methods for sampling and tests for dye intermediates ( <i>first revision</i> )

#### 3 REQUIREMENTS

##### 3.1 Description

The material shall be in the form of white crystalline powder and shall be free from visible impurities.

3.2 The material shall also comply with the requirements given in Table 1.

#### 4 PACKING AND MARKING

##### 4.1 Packing

The material shall be packed in steel drums (*see* IS 2552) or as agreed to between the purchaser and the supplier.

##### 4.2 Marking

Each container shall be securely closed and shall bear legibly and indelibly the following information:

- a) Name of the material;
- b) Indication of the source of manufacture;
- c) Net mass;
- d) Lot or Batch number;
- e) Month and year of manufacture; and
- f) The minimum cautionary note worded as below:  
DANGER! HAZARDOUS SOLID AND VAPOURS RAPIDLY ABSORBED THROUGH SKIN.

##### 4.2.1 BIS Certification Marking

4.2.1.1 Each container may also be marked with the Standard Mark.

4.2.1.2 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the

**Table 1 Requirements for Acetoacet-2-Toluidide**  
(Clauses 3.2, 5.3.1, 5.3.2 and 6.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex of this Standard	Clause of IS
(1)	(2)	(3)	(4)	(5)
i)	Melting point, °C, <i>Min</i>	103	—	9 of IS 5299
ii)	Purity by coupling value, percent by mass, <i>Min</i>	98.0	A	—
iii)	Matter insoluble in sodium hydroxide (dilute) percent by mass, <i>Max</i>	0.2	—	11 of IS 5299
iv)	Impurities:			
	<i>o</i> -Toluidine, percent by mass, <i>Max</i>	0.2	B	—

licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

## 5 SAMPLING

5.1 The method of drawing representative samples of the material shall be as prescribed in 4 of IS 5299.

### 5.2 Number of Tests

Tests for melting point, purity by nitrite value and impurities shall be conducted on each of the individual sample.

Test for determination of solubility description shall be done on composite sample.

### 5.3 Criteria for Conformity

#### 5.3.1 For Individual Samples

The lot shall be declared as conforming to the

requirements of this standard if each of the individual test results satisfies the relevant requirements given in 3.1 and Table 1.

#### 5.3.2 For Composite Samples

For declaring the conformity of the lot to the requirements of matter insoluble and description while tested on the composite sample, the test result shall satisfy the relevant requirements given in Table 1.

## 6 TEST METHODS

6.1 Tests shall be conducted according to the methods prescribed and as indicated in col 4 of Table 1.

### 6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

## ANNEX A

[Table 1, Sl No. (ii)]

### METHODS OF TEST FOR ACETOACET-2-TOLUIDIDE

#### A-1 PURITY BY NITRITE VALUE

##### A-1.0 Outline of the Method

A diazonium solution is prepared by reacting 4-nitroaniline with sodium nitrite solution. It is standardized by titrating against acetoacet-2-toluidide of known purity. The sample of acetoacet-2-toluidide to be tested is then titrated against this standard diazonium solution.

##### A-1.1 Reagents

A-1.1.1 *Hydrochloric Acid* — 30 percent (m/v).

A-1.1.2 *Sodium Nitrite Solution* — 10 percent (m/v).

A-1.1.3 *4-Nitroaniline* — 99 percent pure.

A-1.1.4 *Sodium Hydroxide Solution* — 40 percent (m/v).

A-1.1.5 *Dilute Acetic Acid* — 25 percent (m/v).

A-1.1.6 *Sodium Acetate Solution* — 20 percent (m/v).

A-1.1.7 *H-Acid Indicator Solution* — Dissolve 0.5 g of H-acid in 100 ml of water containing 1g of soda ash.

A-1.1.8 *Acetoacet-2-Toluidide* — Reference sample of known purity.

A-1.1.9 *Tetrazodanisidine Solution* — Take 2 g of dianisidine base in a beaker and dissolve in 7 ml of hydrochloric acid (heat, if necessary upto 50°C). Cool with ice at about 0°C and add immediately 12 ml of sodium nitrite solution and make up the volume to 100 ml with ice cold water. Shake mildly. Test for excess nitrite with starch-iodide test paper. Store this solution in an amber-coloured bottle in a cool place.

A-1.1.10 *Starch-Iodide Test Papers*

##### A-1.2 Procedure

A-1.2.1 *Preparation of Diazonium Solution of 4-Nitroaniline*

Take about 2 g of 4-nitroaniline in a 250-ml glass

beaker containing 5 ml of hydrochloric acid and 100 ml of water, heat upto 60°C to dissolve completely. Cool externally to 0 to 5°C with ice. Then add 10 ml of sodium nitrite solution with stirring. Test for excess of sodium nitrite by spotting on starch-iodide test paper. Allow to stand for 30 min and then repeat the above test. Filter to remove insoluble matter and then make up the filtrate to 200 ml in a volumetric flask. Store the solution in an ice-bath in the dark.

#### A-1.2.2 Standardization of the Diazonium Solution

Weigh accurately 0.700 g of acetoacet-2-toluidide of known purity and dissolve with 1 ml of sodium hydroxide solution in 25 ml of water. Dilute the solution to about 300 ml with water. With stirring add drop-wise dilute acetic acid till pH is 7 which may be judged by using pH paper. Add to the suspension 100 ml of sodium acetate solution. While stirring mechanically add the diazonium solution (see A-1.2.1) from a burette equipped with a jacket through which water is circulating at about 10°C. The burette should be of amber glass to minimize any decomposition of the diazonium salt by light.

Titrate as rapidly as spot test permits. To test for excess acetoacet-2-toluidide place a few drops of titration mixture on a filter paper. About 1 cm away from the edge of the liquid mark, place a few drops of tetrazodanisidine solution. Where the two liquid portions meet on the filter paper, a brown colour will develop if excess of acetoacet-2-toluidide is present. Similarly try with H-acid indicator to test the excess

of diazonium salt. If a pink colour develops at the junction, excess of diazonium solution is indicated.

Initially add diazonium solution in 1 to 2 ml portions, testing titration mixture after each addition for excess of acetoacet-2-toluidide and diazonium salt. As the end-point approaches, diazonium standard solution should be added in portions of 0.2 ml. The end-point is reached when no reddish brown colour is given with tetrazodanisidine solution and no pink colour or very faint colour is given with H-acid indicator. Note the volume of diazonium solution required for titration.

A-1.2.3 Repeat the titration as prescribed in A-1.2.2 using the test sample of acetoacet-2-toluidide.

#### A-1.3 Calculation

A-1.3.1 Normality of diazonium solution 
$$N = \frac{P \times 0.7}{V \times 19.12}$$

where

$P$  = purity (percent by mass) of the known sample; and

$V$  = volume, in ml, of diazonium solution required for the known sample.

A-1.3.2 Purity by coupling value, percent by mass 
$$= \frac{V_1 \times N \times 19.12}{0.7}$$

where

$V_1$  = volume, in ml, of diazonium solution required for the test sample, and

$N$  = normality of diazonium solution.

## ANNEX B

[Table 1, Sl No. (iv)]

### THIN LAYER CHROMATOGRAPHIC ANALYSIS FOR DETERMINATION OF IMPURITIES

#### A-1 GENERAL

Impurities are determined by thin layer chromatography. Reference may be made to IS 5299 for details of TLC test method to be followed. However, necessary details of test conditions are given below for guidance only:

- |   |   |   |
|---|---|---|
| a) Product name                           | : | Acetoacet-2-Toluidide                               |
| b) Sample solution (on 100 percent basis) | : | 1 percent in methanol                               |
| c) Application/sample volume for spotting | : | 5 µl (for sample)<br>2 µl and 4 µl (for impurities) |
| d) Standard                               | : | Reference standard                                  |
| e) Test substance for impurities          | : | <i>o</i> -Toluidine (0.05 percent in methanol)      |



f) Plate type	:	Silica gel G	
g) Eluent	:	Benzene	
h) Elution time	:	25 min	
j) Temperature	:	25 ± 5°C	
k) Detection spray	:	<sup>1)</sup> Diazotize and couple with American base solution	
m) Evaluation	:	Semi-quantitative	
n) Approximate R <sub>f</sub> value—Main band	:	Acetoacet-2-Toluidide	: R <sub>f</sub> 0.5
—Impurities	:	<i>o</i> -Toluidine	: R <sub>f</sub> 0.7

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<sup>1)</sup> American base solution : N-1 (Naphyl) ethylenediamine—Di hydrochloride 1 percent solution in (9:1) Methanol : Water.

**ANNEX C***(Foreword)***COMMITTEE COMPOSITION****Dyes Intermediate Sectional Committee, PCD 11**

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<i>Member Secretary</i>	
DR (SHRIMATI) VIJAY MALIK Director (PCD), BIS	

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This Indian Standard has been developed from Doc : No. PCD 11 (2024).

**Amendments Issued Since Publication**

Amend No.	Date of Issue	Text Affected

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